

1211/8

PATENT
Case 4233C3

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: Donald B. Appleby et al : Paper No. _____
Serial No.: 08/360,184 : Group Art Unit: 1211
Filed: December 20, 1994 : Examiner: E. White
Re: Polyol Polyester Synthesis

Box 400 Amendment
Assistant Commissioner for Patents
Washington, D. C. 20231

RECEIVED

APR 20 1999

MATRIX CUSTOMER
SERVICE CENTER

Dear Sir:

Transmitted herewith is an Amendment and Request for Interference Pursuant to 37 C.F.R. §1.607 in the above-identified application.

[X] Also attached: Associate Power of Attorney

Showing Under 37 C.F.R. 1.608(b) in Support of Request for Interference
Under 37 C.F.R. 1.607

Declaration Under 37 C.F.R. § 1.608 of Steven R. Alexander
Declaration Under 37 C.F.R. § 1.608 of Robert B. Aylor
Declaration Under 37 C.F.R. § 1.608 of Roger S. Berger
Declaration Under 37 C.F.R. § 1.608 of Patrick J. Corrigan
Declaration Under 37 C.F.R. § 1.608 of Diane D. Farris
Declaration Under 37 C.F.R. § 1.608 of Pamela D. Hardy
Declaration Under 37 C.F.R. § 1.608 of Gene P. Hawkins
Declaration Under 37 C.F.R. § 1.608 of Ju-Nan Kao
Declaration Under 37 C.F.R. § 1.608 of Ephraim L. Kelly
Declaration Under 37 C.F.R. § 1.608 of David R. Loggains
Declaration Under 37 C.F.R. § 1.608 of Michael W. McIntosh
Declaration Under 37 C.F.R. § 1.608 of Milton C. McMullen
Declaration Under 37 C.F.R. § 1.608 of Harold R. McPeck
First Declaration Under 37 C.F.R. § 1.608 of Scott D. Pearson
Second Declaration Under 37 C.F.R. § 1.608 of Scott D. Pearson
Declaration Under 37 C.F.R. § 1.608 of Richard G. Schafermeyer
Declaration Under 37 C.F.R. § 1.608 of Carol L. White
Declaration Under 37 C.F.R. § 1.608 of K. Michael Wieser
Exhibits 1-61

APR 21 1999

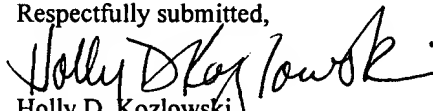
GROUP 1

The fee has been calculated as shown below:

	NO. OF CLAIMS	HIGHEST PREVIOUS PAID FOR	EXTRA CLAIMS	RATE	FEE
Total Claims	76	40	36	x \$18 =	\$648.00
Independent Claims	9	3	6	x \$78 =	\$468.00
TOTAL FEE DUE					\$1,116.00

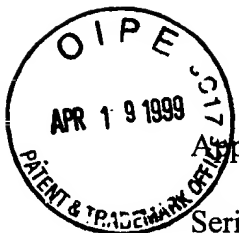
- [X] A check in the amount of \$1,116.00 is enclosed.
- [] Please charge my Deposit Account No. 04-1133 in the amount of \$0.
- [x] The Commissioner is hereby authorized to charge payment of any additional fees associated with this communication or credit any overpayment, to Deposit Account No. 04-1133, including any filing fees under 37 CFR 1.16 for presentation of extra claims and any patent application processing fees under 37 CFR 1.17.

Respectfully submitted,


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434131.01

PATENT
CASE 4233C3



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: Donald B. Appleby et al : Group Art Unit: 1211
Serial No.: 08/360,184 : Examiner: E. White
Filed: December 20, 1994 :
For: Polyol Polyester Synthesis

#31
5-13-99
R. D. Hume

**SHOWING UNDER 37 C.F.R. §1.608(b) IN SUPPORT OF
REQUEST FOR INTERFERENCE UNDER 37 C.F.R. §1.607**

Box Fee Amendment
Assistant Commissioner of Patents
Washington, DC 20231

Dear Sir:

Appleby et al submit herewith an Amendment and Request for Interference Pursuant to 37 C.F.R. §1.607, requesting that an interference be declared between the present application and the Buter U.S. Patent No. 5,043,438 (the Buter patent). The present Showing, together with the Declaration and Exhibits submitted herewith, demonstrate that Appleby et al are *prima facie* entitled to a judgement relative to the patentee, Buter. The basis upon which Appleby et al are entitled to a judgement relative to the patentee, Buter, is priority of invention.

As will be explained with particularity below, the evidence submitted herewith comprising Declarations by the Appleby et al co-inventor Scott D. Pearson and by corroborating witnesses, supported by exhibits comprising documentary evidence, sets forth a factual description of acts and circumstances performed or observed by the Declarants which collectively establish by a

preponderance of evidence that Appleby et al reduced to practice the invention corresponding to the count set forth in the Amendment and Request for Interference pursuant to 37 C.F.R. §1.607 at least as early as January 20, 1989. Moreover, Declarations by the Appleby et al co-inventors Scott D. Pearson, Patrick J. Corrigan, Ju-Nan Kao and Richard G. Shafermeyer and by additional witnesses and supporting exhibits demonstrate by a preponderance of evidence that the Appleby et al invention was not abandoned, suppressed or concealed between the reduction to practice in January 1989 and the filing of the original Appleby et al application in September 1990.

The evidence therefore *prima facie* entitles Appleby et al to judgement on priority with respect to either the Buter patent actual U.S. filing date of February 9, 1990 or either of the Buter patent's alleged effective U.S. filing dates under 35 U.S.C. §119 of February 16, 1989 or November 20, 1989 (although Appleby et al do not concede that Buter is entitled to either alleged effective U.S. filing date).

I. THE APPLEBY ET AL INVENTION CORRESPONDING TO THE PROPOSED COUNT WAS COMPLETED BY JANUARY 20, 1989

The Appleby et al invention corresponding to the proposed count was reduced to practice at least as early as January 20, 1989.

A. Requirements of Showing Based on Priority of Invention

According to 37 C.F.R. § 1.608(b):

When the effective filing date of an application is more than three months after the effective filing date of a patent, the applicant, before an interference will be declared, shall file evidence which may consist of patents or printed publications, other documents, and one or more affidavits which demonstrate that applicant is *prima facie* entitled to a judgment relative to the patentee and an explanation stating with particularity the basis upon which the applicant is *prima facie* entitled to the judgment. Where the basis upon which an applicant is entitled to judgment relative

to a patentee is priority of invention, the evidence shall include affidavits by the applicant, if possible, and one or more corroborating witnesses, supported by documentary evidence, if available, each setting out a factual description of acts and circumstances performed or observed by the affiant, which collectively would *prima facie* entitle the applicant to judgment on priority with respect to the effective filing date of the patent.

To establish a *prima facie* case of priority, the junior party is required to prove at least so much of his case as would entitle him to an award of priority if the senior party were to rely only on his filing date and were not to rebut any of the junior party's case. *Hahn v. Wong*, 13 U.S.P.Q.2d 1313, 1317 (Fed. Cir. 1989). Specifically:

“[T]he party who files later is ‘required to establish reduction to practice before the filing date of the party who filed first, or conception before that date coupled with reasonable diligence from just before that date to the filing date of the party who files later.’” *Hahn v. Wong, Id.*, citing *Oka v. Youssefyeh*, 7 U.S.P.Q.2d 1169, 1172 (Fed. Cir. 1988).

An actual reduction to practice of an invention in an interference must include every essential limitation of the count. *Correge v. Murphy*, 217 USPQ 753, 755 (Fed. Cir. 1983). To establish a reduction to practice of a method count, it is necessary to show that each step of the method was performed and that the product produced by the method was satisfactory for its intended use. *English v. Ausnit*, 38 U.S.P.Q.2d 1625, 1634 (BPAI 1993) (citations omitted). The quantum of proof required to establish priority in an interference with an issued patent is a preponderance of evidence. *Bosies v. Benedict*, 30 U.S.P.Q.2d 1862, 1864 (Fed. Cir. 1994). The Declarations and supporting documentary evidence submitted herewith demonstrate by a preponderance of evidence Appleby et al.’s reduction to practice of every essential limitation of the proposed count prior to the filing date of the Buter patent.

Only an inventor’s testimony needs corroboration, *Holmwood v. Sugavanam*, 20 U.S.P.Q.2d 1712, 1715 (Fed. Cir. 1991). Moreover:

“Over-the-shoulder observation of every step is not necessary to satisfy the corroboration rule where there is independent circumstantial evidence of a reduction to practice...This is especially true where...the activities in question were apparently part of an organized program of research.” *Nashef v. Pollock*, 4 U.S.P.Q.2d 1631, 1636 (BPAI 1987) (citations omitted).

Thus, corroborative testimony does not have to be an actual witnessing of the reduction to practice by one who understands what is going on in order to be adequate, *Berges v. Gottstein*, 205 U.S.P.Q. 691, 695 (CCPA 1980). Facts set forth in affidavits can trace a highly organized procedure routinely practiced for identifying, preserving and testing newly synthesized compounds by a research team. *Id.*, at 694. The actual witnessing of such highly organized procedures by another individual is not required.

Additionally, a “rule of reason” is applied when reviewing the sufficiency of evidence of a reduction to practice, wherein the realities of technical operations in modern day research laboratories cannot be ignored, *Holmwood v. Sugarvana*, *supra* at 1714. This rule requires the Patent and Trademark Office to examine, analyze and evaluate reasonably all pertinent evidence when weighing the credibility of an inventor’s story. The “rule of reason” recognizes that a junior technician performing perfunctory tasks under the supervision of a senior scientist is not generally necessary to verify the reliability of evidence about scientific methods or data; in the absence of indicia calling into question the trustworthiness of the senior scientist’s testimony, the “rule of reason” permits the Board to rely on the trained supervisor’s testimony to ascertain scientific methods or results, *Holmwood v. Sugarvanam*, *Id.* The testimony of the coinventor Scott D. Pearson is sufficiently corroborated by noninventor Declarants and supporting documentary evidence to establish by a preponderance of evidence Appleby et al’s reduction to practice before the filing date of the Buter patent.

B. The Appleby et al Invention Reduction to Practice

From January 18, 1989 to January 27, 1989, a pilot plant continuous sucrose polyester production process within the scope of the proposed count was performed under the direction and control of the Appleby et al co-inventor Scott D. Pearson using the pilot plant located in Building 96 of The Procter & Gamble Company's Ivorydale plant. This pilot plant continuous sucrose polyester production process was designated the P90117 pilot plant continuous process. The First Declaration Under 37 C.F.R. 1.608 of Scott D. Pearson (the First Pearson Declaration) by the coinventor Pearson, together with the respective corroborating Declarations Under 37 C.F.R. 1.608 of non-inventors Diane Farris, Pamela D. Hardy, Gene P. Hawkins, David R. Loggains, Michael W. McIntosh, Harold R. McPeck, Milton C. McMullen, Carol L. White and K. Michael Wieser and Exhibits Nos. 1-9 identified in those Declarations, all of which are submitted herewith, set forth a factual description of acts and circumstances performed or observed by the Declarants which collectively establish by a preponderance of evidence that Appleby et al reduced to practice the invention corresponding to the proposed count during the P90117 pilot plant continuous process on January 20, 1989.

1. The Proposed Count

The proposed count set forth in the Amendment and Request for Interference Pursuant to 37 C.F.R. §1.607 (the proposed count) is as follows:

A process for the synthesis of polyol fatty-acid esters by reacting a polyol and a fatty-acid lower-alkyl ester under substantially solvent free conditions in the presence of a catalyst and an emulsifier, the process comprising:

an initial reaction stage which is carried out under such conditions that the reaction mixture in said initial stage is in steady-state, with continuous introduction of reactants comprising polyol and fatty-acid lower-alkyl ester, and continuous

removal of products comprising reaction mixture having a degree of esterification of about 1% or more and volatile alcohol formed during the initial reaction stage of the synthesis, and

and one or more subsequent reaction stages in which the reaction mixture from said initial stage is further reacted to said polyol fatty-acid esters.

2. The P90117 Pilot Plant Continuous Process Contained All Essential Limitations of the Proposed Count

As described by the Appleby et al coinventor Declarant Pearson, and corroborated by the respective noninventor Declarants Farris, Hardy, Hawkins, Loggains, McIntosh, McPeck, McMullen, White and Wieser, the P90117 pilot plant continuous process comprised all of the essential limitations of the proposed count. The essential limitations of the proposed count and the corresponding activity and supporting Declarations are summarized in Table 1 and discussed in detail hereafter:

Table 1: Comparison of Essential Limitations of Proposed Count to P90117 Pilot Plant Continuous Process

Proposed Count	P90117 Pilot Plant Continuous Process
A process for the synthesis of polyol fatty-acid esters by reacting a polyol and a fatty-acid lower-alkyl ester under substantially solvent free conditions in the presence of a catalyst and an emulsifier,	Polyol fatty acid esters (sucrose fatty acid polyesters) were synthesized in the P90117 pilot plant continuous process by reacting a polyol (sucrose) and a fatty-acid lower-alkyl ester (fatty acid methyl ester) under substantially solvent free conditions in the presence of a transesterification catalyst (potassium carbonate) and an emulsifier (potassium soap). First Pearson Declaration, ¶¶ 9 and 10; Farris Declaration, ¶¶ 9 and 10.

the process comprising: an initial reaction stage which is carried out under such conditions that the reaction mixture in said initial stage is in steady-state,	The P90117 pilot plant continuous process comprised an initial reaction stage, reactor R600, carried out under such conditions that the reaction mixture in reactor R600 was in steady state, with mass-balanced ingoing reactant streams and outgoing product streams. First Pearson Declaration, ¶¶ 9 and 11-21; Farris Declaration, ¶¶ 9 and 11-21.
with continuous introduction of reactants comprising polyol and fatty-acid lower-alkyl ester,	The continuous ingoing reactant streams comprised polyol (sucrose) and fatty acid lower alkyl ester (fatty acid methyl ester). First Pearson Declaration, ¶¶ 10-12; Farris Declaration, ¶¶ 10-12.
and continuous removal of products comprising reaction mixture having a degree of esterification of about 1% or more and volatile alcohol formed during the initial reaction stage of the synthesis, and	The continuous outgoing product streams comprised reaction mixture having a polyol conversion (sucrose conversion) of 1% or more and lower alkyl alcohol (methanol) formed during the initial stage of the synthesis. First Pearson Declaration, ¶¶ 13, 15-17 and 20; Farris Declaration, ¶¶ 13, 15-17 and 20.
one or more subsequent reaction stages in which the reaction mixture from said initial stage is further reacted to said polyol fatty-acid esters.	The process further comprised a subsequent reaction stage (reactors R601-R606) in which the reaction mixture from the first reactor R600 was further reacted to the polyol fatty acid esters (sucrose fatty acid esters) in the subsequent reaction stages. First Pearson Declaration, ¶¶ 7-9 and 22; Farris Declaration, ¶¶ 7-9 and 22.

3. The Pilot Plant Continuous Process

The P90117 pilot plant continuous sucrose polyester production process was conducted using the pilot plant located in Building 96 of The Procter & Gamble Company's Ivorydale plant (First Pearson Declaration, ¶5; Farris Declaration, ¶4). The P90117 pilot plant continuous process was performed under the direction and control of Scott D. Pearson, a Group Leader on the sucrose polyester synthesis project (First Pearson Declaration, ¶¶ 4-5; Farris Declaration, ¶¶ 3-4). Diane D.

Farris, Section Head on the sucrose polyester synthesis project, was also present during and observed and reviewed the P90117 pilot plant continuous process (Farris Declaration, ¶5). Carol L. White supervised technicians monitoring various operating parameters during the P90117 pilot plant continuous process (White Declaration, ¶5), while Pamela D. Hardy, Gene R. Hawkins, David R. Loggains, Milton C. McMullen, Harold R. McPeck and K. Michael Wieser worked as technicians on the P90117 pilot plant continuous process (Hardy Declaration, ¶3; Hawkins Declaration, ¶3; Loggains Declaration, ¶2; McMullen Declaration, ¶2; McPeck Declaration, ¶2; Wieser Declaration, ¶2). During the P90117 pilot plant continuous process, the operation of the continuous sucrose polyester production equipment, sample collection, analysis of collected samples, and data recordation and review were performed under Mr. Pearson's direction and control in accordance with established procedures (First Pearson Declaration, ¶5; Farris Declaration, ¶4).

Exhibit 1 comprises an accurate copy of page 4.1 of a manual entitled "Continuous Pilot Plant Start-Up Manual" authored on or about September 26, 1986 by Michael W. McIntosh, a technician working on the sucrose polyester synthesis project (McIntosh Declaration, ¶¶ 2-3) and is a general schematic diagram of the reactors employed in the pilot plant equipment used in the P90117 pilot plant continuous process (First Pearson Declaration ¶7; Farris Declaration, ¶7). Exhibit 2 is an accurate copy of the P90117 Experimental Test Plan dated January 12, 1989, prepared on or about January 12, 1989 by Mr. Pearson, and sets forth the conditions under which the P90117 pilot plant continuous process was run from January 18-January 22, 1989 (Pearson Declaration ¶8; Farris Declaration, ¶8). As set forth on page 2 of Exhibit 2, the P90117 pilot plant continuous process employed seven continuous stirred tank reactors, reactors R600-R606, respectively, in series (Pearson Declaration, ¶8; Farris Declaration, ¶8).

4. Synthesis of Polyol Fatty-Acid Esters by Reacting a Polyol and a Fatty-Acid Lower Alkyl Ester Under Substantially Solvent Free Conditions and in the Presence of a Catalyst and an Emulsifier

The P90117 pilot plant continuous process comprised a process for the synthesis of polyol fatty-acid esters by reacting a polyol and a fatty-acid lower-alkyl ester under substantially solvent free conditions in the presence of a catalyst and an emulsifier. As defined by both the Buter patent and the Appleby et al application, a polyol is a compound which comprises at least four free hydroxyl groups (Buter patent, column 1, line 15; Appleby et al application, page 4, lines 33-34), for example, sucrose (Buter patent, column 1, line 25; Appleby et al application, page 24). Both the Buter patent and the Appleby et al application disclose that emulsifiers include soaps (Buter patent, column 4, line 30; Appleby et al application, page 11, lines 7-8), and that transesterification catalysts include basic catalysts such as potassium carbonate (Buter patent, column 3, line 68-column 4, line 1; Appleby et al application, page 11, lines 36-37). Buter discloses that suitable fatty-acid lower-alkyl esters are fatty-acid esters of lower alcohols, such as methanol (column 3, lines 38-41), while Appleby et al teach that fatty acid esters include esters of volatile alcohols, such as methanol (page 5, lines 29-30). Therefore, according to both Buter and Appleby et al, fatty-acid lower-alkyl esters include fatty acid methyl esters.

Sucrose fatty acid polyesters were synthesized in the P90117 pilot plant continuous process by reacting polyol, namely sucrose, and fatty-acid lower-alkyl ester, namely fatty acid methyl ester, in the presence of catalyst comprising potassium carbonate and emulsifier comprising potassium soap (First Pearson Declaration, ¶¶ 9-11, 15, 16 and 22; Farris Declaration, ¶¶ 9-11, 15, 16 and 22). More particularly, as described at pages 2-4 of Exhibit 2, a sucrose feed batch comprising sucrose, fatty acid methyl ester and potassium soap emulsifier, and a catalyst batch comprising potassium

carbonate catalyst and fatty acid methyl ester were the in-going feed streams to the first reactor, reactor R600, employed in the P90117 pilot plant continuous process (First Pearson Declaration, ¶10; Farris Declaration, ¶10). Exhibits 3 and 4 comprise accurate copies of the control records evidencing the components employed in forming the sucrose feed batch and the catalyst batch, respectively, for the P90117 pilot plant continuous process (Wieser Declaration, ¶4). Exhibits 3 and 4 demonstrate that the feed materials for the P90117 pilot plant continuous process were substantially solvent free whereby the polyol fatty acid ester synthesis reaction was conducted under substantially solvent-free conditions (Wieser Declaration, ¶4; First Pearson Declaration, ¶10; Farris Declaration, ¶10).

Exhibit 8 comprises accurate copies of data sheets from the P90117 pilot plant continuous sucrose polyester process which set forth determined average degree of sucrose esterification (\bar{I}) and area percentages of sucrose monoester, diester, triester, tetraester, pentaester, hexaester, heptaester and octaester, respectively, in the sucrose polyester, in reactor recirculation loop samples (Hardy Declaration, ¶¶ 5-6; Hawkins Declaration, ¶¶ 5-6). The reactor recirculation loop samples are representative of the respective reactor out-going streams (First Pearson Declaration, ¶7; Farris Declaration, ¶7). Exhibit 8 demonstrates that octaester was present in out-going streams of Reactors R603, R604 and R605 during the P90117 pilot plant continuous process (First Pearson Declaration, ¶22; Farris Declaration, ¶22). Therefore, during the P90117 pilot plant continuous process, polyol fatty acid esters (sucrose fatty acid polyesters, including octaesters) were synthesized by reacting polyol (sucrose) and fatty acid lower alkyl ester (fatty acid methyl ester) under substantially solvent free conditions and in the presence of a transesterification catalyst (potassium carbonate) and an emulsifier (potassium soap).

5. The Reaction Mixture in the Initial Stage was in Steady State with Continuous Introduction of Reactants and Continual Removal of Products

The P90117 pilot plant continuous process comprised an initial reaction stage which was carried out under such conditions that the reaction mixture in the initial stage was in steady-state, with continuous introduction of reactants comprising polyol and fatty-acid lower-alkyl ester, and continuous removal of products comprising reaction mixture having a degree of esterification of about 1% or more and volatile alcohol formed during the initial reaction stage of the synthesis.

As described above, a general schematic diagram of the reactors employed in the pilot plant equipment used in the P90117 pilot plant continuous process is set forth on page 4.1 of Exhibit 1 (First Pearson Declaration ¶7; Farris Declaration ¶7). The pilot plant equipment comprised a series of seven continuous stirred tank reactors (CSTRs) numbered R600 through R606, respectively (First Pearson Declaration, ¶¶ 7-8; Farris Declaration, ¶¶ 7-8). Flow to each reactor was either raw material feed and/or flow controlled reactor product from the previous reactor, and discharge from each reactor was by level control (First Pearson Declaration, ¶¶ 7 and 13; Farris Declaration, ¶¶ 7 and 13). The first reactor, R600, comprised an initial reaction stage (First Pearson Declaration, ¶8; Farris Declaration, ¶8).

a. Continuous Introduction of Reactants Comprising Polyol and Fatty-acid Lower-alkyl Ester

As described at pages 2-4 of Exhibit 2, the P90117 Experimental Test Plan, a sucrose feed batch comprising sucrose, fatty acid methyl ester and potassium soap emulsifier, and a catalyst batch comprising potassium carbonate catalyst and fatty acid methyl ester were the in-going feed streams to the first reactor, R600, in the P90117 pilot plant continuous process (First Pearson Declaration,

¶¶ 9-10; Farris Declaration, ¶¶ 9-10). As set forth above, Exhibits 3 and 4 comprise accurate copies of the control records evidencing the components employed in forming the sucrose feed batch and the catalyst batch, respectively, for the P90117 pilot plant continuous process (Wieser Declaration, ¶4). Under Mr. Pearson's direction and control and Mr. Wieser's supervision and control, potassium soap was prepared in a reactor 001 by the reaction of fatty acid ester (I-1) and potassium hydroxide in the presence of methanol, and the methanol was evaporated off. Fatty acid methyl ester (methyl ester blend) and sucrose were then added to the remaining potassium soap to form the sucrose feed batch. Under Mr. Pearson's further direction and control and Mr. Wieser's further supervision and control, potassium carbonate and fatty acid methyl ester (ester blend Y) were mixed in reactor 501 to form the catalyst batch.

The sucrose feed batch components and the catalyst batch components as set forth in Exhibits 3 and 4 are presented herein, respectively, in Tables 2 and 3:

Table 2: Sucrose Feed Batch Components

Component	Pounds per batch
KOH*	115
Fatty Acid Ester (I-1)*	600
Methanol**	1200
Fatty Acid Methyl Ester	3700
Sucrose	800

* The fatty acid ester (I-1) and KOH were reacted prior to addition of methyl ester and sucrose to form the potassium soap emulsifier.

** The methanol was evaporated off prior to the sucrose addition.

Table 3: Catalyst Batch Components

Component	Pounds per batch
Potassium Carbonate	250
Fatty Acid Methyl Ester	750

(Wieser Declaration, ¶4; First Pearson Declaration, ¶10; Farris Declaration, ¶10).

As described at pages 2-3 of Exhibit 2, during the P90117 pilot plant continuous process, the sucrose feed batch and the catalyst batch were fed to the first reactor (R600), and after start-up and during the period of time from 2:00 a.m. to 13:00 (1:00 p.m.) on January 20, 1989, all reactors in the P90117 pilot plant continuous process were full and feeding forward (First Pearson Declaration, ¶11; Farris Declaration, ¶11) .

Exhibit 5 comprises accurate copies of the process operating log sheets for operation of the P90117 pilot plant continuous process on January 20, 1989 for the time period of from 2:00 am to 13:00 (1:00 pm) (White Declaration, ¶5). Under Mr. Pearson's direction and control and Ms. White's supervision, technicians monitoring the various operating parameters of the pilot plant system observed and entered the operating parameters on the log sheets of Exhibit 5 on an hourly basis and in accordance with established procedures (White Declaration, ¶5; First Pearson Declaration, ¶12; Farris Declaration, ¶12). More specifically, the operating parameters comprising reactor pressure, reactor temperature, slurry feed batch flowrate, confirmation of operation of catalyst injector and injection cycle (dump time--the period between successive catalyst injections) for reactor R600 as set forth in Exhibit 5 are presented herein in Table 4:

Table 4: Operating Parameters for Reactor R600 on January 20, 1989

Time	Reactor Pressure (mm/Hg)	Reactor Temperature (°F)	Slurry Feed Batch Flowrate (pounds/hour)	Catalyst Batch Operating, 0.136 Pounds/ Injection	Injection Cycle (min.)
2:00 a.m.	15.1	275.1	55.2	yes	11
3:00 a.m.	14.9	274.9	55.0	yes	11
4:00 a.m.	15.3	275.3	54.7	yes	11
5:00 a.m.	15.2	275.1	54.9	yes	11
6:00 a.m.	15.0	275.0	54.9	yes	11
7:00 a.m.	14.9	275.1	54.8	yes	11
8:00 a.m.	15.2	275.7	54.9	yes	11
9:00 a.m.	15.4	275.1	54	yes	11
10:00 a.m.	15.1	275.4	55.3	yes	11
11:00 a.m.	15.1	275.1	55	yes	11
12:00 p.m.	14.5	275.4	55	yes	11
13:00 (1:00) p.m.	14.9	275.3	55.2	yes	11

(First Pearson Declaration, ¶12; Farris Declaration, ¶12).

Exhibit 5, and the data reproduced from Exhibit 5 and set forth in Table 4, demonstrates that the reactor pressure, reactor temperature, slurry feed batch flowrate, and catalyst batch injection rate (the rate at which the catalyst feed mixture was injected) for the initial reactor, reactor R600, were substantially constant over the time period of from 2:00 a.m. to 13:00 (1:00 p.m.) on January 20, 1989, and particularly that there was a continuous introduction of reactants comprising polyol (sucrose) and fatty acid lower alkyl ester (fatty acid methyl ester) into the initial reaction stage at a constant rate (First Pearson Declaration, ¶12; Farris Declaration, ¶12).

b. Continuous Removal of Products

Exhibit 6 comprises an accurate copy of the Process Log Notebook for the P90117 pilot plant continuous process for the days of January 17, 1989 through January 20, 1989 and indicates that the level on the first reactor R600 was set at 8" on January 18, 1989, whereby once reactor R600 was filled, the contents of the reactor were maintained at a constant level throughout the reaction by level controlled discharge of product from the reactor (First Pearson Declaration, ¶13; Farris Declaration, ¶13). Since there was a continuous introduction of reactants into the initial reaction stage in the first reactor R600 at a constant rate during the time period of from 2:00 a.m. to 13:00 (1:00 p.m.) on January 20, 1989, and since the first reactor R600 was not accumulating mass owing to the level control discharge, there was also a continuous removal of product from the initial reaction stage, i.e., the first reactor R600, over the same time period (*Id.*). Thus, the reaction mixture in the first reaction stage, i.e., the first reactor R600, was overall mass-balanced during the time period of from 2:00 a.m. to 13:00 (1:00 p.m.) on January 20, 1989 (*Id.*).

c. Steady State Reaction Mixture

The initial reaction stage in reactor R600 was carried out under such conditions that the reaction mixture in the initial reaction stage was in steady state. As set forth by Felder et al, *Elementary Principals of Chemical Processes* (Wiley 1978), p. 82 (Appendix B to the Amendment and Request for Interference Pursuant to 37 C.F.R. §1.607 submitted herewith), in a continuous process, the inputs and outputs flow continuously through the duration of the process and, if all the variables in a process do not change with time, except possibly for minor fluctuations about constant mean values, the process is said to be operating at steady state. As demonstrated above, there was a continuous introduction of substantially constant reactants to the initial reaction stage, reactor

R600, continuous removal of products from reactor R600, and substantially constant operating parameters in reactor R600. As demonstrated hereafter, the composition of the product stream from reactor R600 was also substantially constant, whereby reactor R600 was operating at steady state.

Exhibit 7 comprises accurate copies of data sheets which set forth determined weight percentages of potassium carbonate catalyst (as carbonate) and potassium soap (as soap) in recirculation loop samples from the first reactor R600 (Loggains Declaration, ¶8; McPeck Declaration, ¶8; Wieser Declaration, ¶6). Throughout the P90117 pilot plant continuous process, samples were taken from the respective recirculation loops of the reactors R600-R606; these samples were representative of the respective reactor's out-going product stream (First Pearson Declaration, ¶7; Farris Declaration, ¶7). More specifically, the weight percentages of potassium carbonate catalyst (as carbonate) and potassium soap (as soap) in the R600 recirculation loop samples obtained at 2:00 a.m., 6:00 a.m., 9:00 a.m., and 13:00 (1:00 p.m.) on January 20, 1989 as set forth in Exhibit 7 are presented herein in Table 5:

Table 5: Weight Percentages of Carbonate and Soap in R600 Samples on January 20, 1989

Time	% Carbonate	% Soap
2:00 a.m.	0.24	12.41
6:00 a.m.	0.13	12.68
9:00 a.m.	0.18	12.69
13:00 (1:00 p.m.)	0.19	12.65

(First Pearson Declaration, ¶14; Farris Declaration, ¶14).

Exhibit 7 demonstrates that the weight percentages of catalyst (as carbonate) and emulsifier (as soap) in the out-going stream from reactor R600 were at substantially constant levels during the

P90117 pilot plant continuous process on January 20, 1989 for the time period of from 2:00 a.m. to 13:00 (1:00 p.m.) (First Pearson Declaration, ¶14; Farris Declaration, ¶14).

Exhibit 8 comprises accurate copies of data sheets which set forth determined area percentages of sucrose monoester, diester, triester, tetraester, pentaester, hexaester, heptaester and octaester, respectively, in the sucrose ester in recirculation loop samples from the first reactor R600 (Hardy Declaration, ¶6; Hawkins Declaration, ¶6). As noted above, the reactor recirculation loop samples are representative of the reactor's out-going product stream (First Pearson Declaration, ¶7; Farris Declaration, ¶7). More specifically, the respective ester area percentages in the sucrose ester in the R600 samples obtained at 4:00 a.m., 7:00 a.m., 10:00 a.m. and 13:00 (1:00 p.m.) on January 20, 1989 as set forth in Exhibit 8 are presented herein in Table 6:

**Table 6: Area Percentages of Respective Esters in
Sucrose Ester in R600 Samples on January 20, 1989**

Time	Monoester	Diester	Triester	Tetraester	Pentaester	Hexaester	Heptaester	Octaester
4:00 a.m.	10.4	26.3	35.3	22.1	5.9	0	0	0
7:00 a.m.	10.4	27.8	36.6	22.1	3.2	0	0	0
10:00 a.m.	8.2	27.5	35.6	22.5	6.2	0	0	0
13:00 (1:00 p.m.)	9.9	27.1	36.6	20.9	5.5	0	0	0

(First Pearson Declaration, ¶15; Farris Declaration, ¶15).

Exhibit 8 demonstrates that the area percentages of sucrose monoester, diester, triester, tetraester, pentaester, hexaester, heptaester and octaester, respectively, in the sucrose ester in the out-going stream from reactor R600 were at substantially constant levels during the P90117 pilot plant

continuous process on January 20, 1989 for the time period of from 2:00 a.m. to 13:00 (1:00 p.m.) (First Pearson Declaration, ¶15; Farris Declaration, ¶15).

Exhibit 8 further comprises accurate copies of data sheets which set forth determined I-bar values representing an average degree of esterification of the sucrose ester, based on mole fractions, in recirculation loop samples from the first reactor R600 (Hardy Declaration, ¶¶ 5, 6 and 9; Hawkins Declaration, ¶¶ 5, 6 and 9), with the recirculation loop samples being representative of the reactor's out-going product stream (First Pearson Declaration, ¶7; Farris Declaration, ¶7). More specifically, the I-bar values of the reactor R600 samples as set forth in Exhibit 8 obtained at 4:00 a.m., 7:00 a.m., 10:00 a.m. and 13:00 (1:00 p.m.) on January 20, 1989 are presented herein in Table 7:

**Table 7: I-Bar Degree of Esterification for
R600 Samples on January 20, 1989**

Time	I-Bar Degree of Esterification
4:00 a.m.	2.57
7:00 a.m.	2.53
10:00 a.m.	2.63
13:00 (1:00 p.m.)	2.56

(First Pearson Declaration, ¶16; Farris Declaration, ¶16).

Exhibit 8 demonstrates that the I-bar values of the sucrose ester in the out-going stream from reactor R600 were substantially constant during the P90117 pilot plant continuous process on January 20, 1989 for the time period of from 2:00 a.m. to 13:00 (1:00 p.m.), indicating that a substantially constant average degree of esterification was obtained in reactor R600 over the same time period (First Pearson Declaration, ¶16; Farris Declaration, ¶16). The I-bar average degree of esterification for each of the reactor samples was in excess of 2, indicating that, on average, in excess

of 2 of the 8 esterifiable hydroxyl groups per sucrose molecule had been esterified, whereby the degree of esterification of the out-going stream from the initial reaction stage, reactor R600, was in excess of 25% (First Pearson Declaration, ¶16; Farris Declaration, ¶16).

Exhibit 9 comprises accurate copies of data sheets which set forth determined weight percentages of unreacted sucrose in recirculation loop samples obtained from the first reactor R600 (Loggains Declaration, ¶5; McMullen Declaration, ¶5; McPeck Declaration, ¶5). As noted above, the reactor recirculation loop samples are representative of the reactor's out-going product stream (First Pearson Declaration, ¶7; Farris Declaration, ¶7). More specifically, the weight percentages of unreacted sucrose in the R600 samples at 2:00 a.m., 6:00 a.m., 7:00 a.m., 8:00 a.m., 9:00 a.m. and 13:00 (1:00 p.m.) on January 20, 1989 as set forth in Exhibit 9 are presented herein in Table 8:

**Table 8: Weight Percentage of Unreacted Sucrose
in R600 Samples on January 20, 1989**

Time	% Sucrose
2:00 a.m.	1.4
6:00 a.m.	1.03
7:00 a.m.	1.47
8:00 a.m.	1.55
9:00 a.m.	1.33
13:00 (1:00 p.m.)	1.39

(First Pearson Declaration, ¶17; Farris Declaration, ¶17).

Exhibit 9 demonstrates that the weight percentage of unreacted sucrose in the out-going stream from reactor R600 was at a substantially constant level during the P90117 pilot plant

continuous process on January 20, 1989 for the time period of from 2:00 a.m. to 13:00 (1:00 p.m.) (First Pearson Declaration, ¶17; Farris Declaration, ¶17).

As discussed above, the data set forth in Exhibit 5 demonstrates that for the time period of 2:00 a.m. to 13:00 (1:00 p.m.) on January 20, 1989, in-going sucrose to reactor R600 (i.e. the sucrose feed batch flow to reactor R600) was at a substantially constant rate, while the data set forth in Exhibit 9 demonstrates that the weight percentage of unreacted sucrose leaving reactor R600 was substantially constant for the same time period. As reactor R600 was mass-balanced and not accumulating mass, and the sucrose polyester synthesis was the only significant reaction in which the sucrose participated in reactor R600, the amount of reacted sucrose in the form of sucrose esters in the product stream leaving reactor R600 was therefore also substantially constant for the same time period (First Pearson Declaration, ¶¶ 12, 17 and 18; Farris Declaration, ¶¶ 12, 17 and 18).

Additionally, the data set forth in Exhibit 8 indicates that for the period of 2:00 a.m. to 13:00 (1:00 p.m.) on January 20, 1989, the area percentage distribution of the respective sucrose esters and the I-bar average degree of esterification in the out-going product stream leaving reactor R600 were substantially constant. Since the amount of sucrose esters in the out-going product stream leaving reactor R600 was also substantially constant for the time period of 2:00 p.m. to 13:00 (1:00 p.m.) on January 20, 1989, and the sucrose-fatty acid methyl ester reaction to form sucrose esters was the only significant reaction occurring in reactor R600, the amounts of the respective individual sucrose esters and unreacted fatty acid methyl ester in the out-going product stream leaving reactor R600 were also substantially constant for the same time period (First Pearson Declaration, ¶¶ 15, 16 and 19; Farris Declaration, ¶¶ 15, 16 and 19).

During the transesterification reaction, sucrose was reacted with fatty acid methyl ester to form sucrose esters and methanol (First Pearson Declaration, ¶20; Farris Declaration, ¶20). Since the amounts of the respective sucrose esters in the out-going product stream leaving reactor R600 were substantially constant for the time period of 2:00 p.m. to 13:00 (1:00 p.m.) on January 20, 1989, and reactor R600 was not accumulating mass, the amount of methanol leaving reactor R600 was also substantially constant for the same time period (First Pearson Declaration, ¶20; Farris Declaration, ¶20).

To summarize, during the P90117 pilot plant continuous process on January 20, 1989, for the time period of from 2:00 a.m. to 13:00 (1:00 p.m.), polyol fatty acid esters were synthesized by reacting sucrose and fatty acid methyl ester under substantially solvent free conditions and in the presence of a transesterification catalyst, potassium carbonate, and an emulsifier, potassium soap. The sucrose and fatty acid methyl ester reactants, the potassium soap emulsifier and the potassium carbonate catalyst were fed to the first reaction zone in the initial reaction stage, i.e., reactor R600, at substantially constant rates, and the operating parameters of reactor R600 were maintained substantially constant. Additionally, during the same time period, the amounts of the respective sucrose esters, the amount of unreacted sucrose, the amount of unreacted fatty acid methyl ester, the amount of methanol by-product, the amount of potassium soap emulsifier and the amount of potassium carbonate catalyst in the out-going product stream leaving reactor R600 were substantially constant. Accordingly, the reaction mixture in R600 was in steady state with continuous introduction of reactants and continuous removal of products (First Pearson Declaration, ¶21; Farris Declaration, ¶21).

d. Continuous Removal of Products Comprising a Reaction Mixture Having a Degree of Esterification of 1% or more and a Volatile Alcohol Formed During the Initial Reaction Stage

The P90117 pilot plant continuous process included the continuous removal of products from the initial reaction stage comprising reaction mixture having a degree of esterification of about 1% or more and volatile alcohol (methanol) formed during the initial reaction stage of the synthesis.

The percentage "degree of esterification" is the percentage of total available hydroxy groups on the polyol which are esterified with a fatty acyl radical (Appleby et al application, page 2, lines 20-23). As discussed above, the "I-bar" value represents an average degree of esterification based on mole fractions (Hardy Declaration, ¶¶ 5 and 9; Hawkins Declaration, ¶¶ 5 and 9). As indicated by the data set forth in Table 5 above, the I-bar value for the out-going product stream from the first reactor, reactor R600, for the period of from 2:00 a.m. to 13:00 (1:00 p.m.) on January 20, 1989, was in excess of 2, indicating that, on average, in excess of 2 of the 8 esterifiable hydroxyl groups per sucrose molecule had been esterified, or, in other words, the percentage degree of esterification of the sucrose ester in the out-going product stream from the initial reaction stage, reactor R600, was in excess of 25% (First Pearson Declaration, ¶16; Farris Declaration, ¶16).

As also discussed above, during the transesterification reaction, sucrose and fatty acid methyl ester reacted to form sucrose esters and methanol (First Pearson Declaration, ¶20, Farris Declaration, ¶20). As the first reactor, reactor R600, was not accumulating mass (First Pearson Declaration, ¶13; Farris Declaration, ¶13), the methanol was continuously removed from the initial reaction stage, reactor R600 (First Pearson Declaration, ¶¶ 20 and 21; Farris Declaration, ¶¶ 20 and 21).

Therefore, the P90117 pilot plant study was performed with continuous removal of products from the initial reaction stage comprising a reaction mixture having a degree of esterification of 1% or more and a volatile alcohol formed during the initial reaction stage of the synthesis.

6. Subsequent Reaction Stages in Which the Reaction Mixture from the Initial Stage was Further Reacted

Finally, the P90117 pilot plant continuous process comprised one or more subsequent reaction stages in which the reaction mixture from the initial stage was further reacted to polyol fatty-acid esters. As set forth in the P90117 Experiment Test Plan (Exhibit 2), the P90117 pilot plant continuous process employed seven continuous stirred tank reactors in series, wherein the first reactor, R600, was an initial reaction stage and subsequent reactors R601-R606, including reactors R603, R604 and R605, the fourth, fifth and sixth reactors, respectively, provided subsequent reaction stages (First Pearson Declaration, ¶¶ 8, 9 and 22; Farris Declaration, ¶¶ 8, 9 and 22).

As described above, Exhibit 8 comprises accurate copies of data sheets which set forth determined area percentages of respective esters in the sucrose ester in reactor recirculation loop samples (Hardy Declaration, ¶6; Hawkins Declaration, ¶6). Exhibit 8 includes data sheets setting forth determined area percentages of octaester in samples from the fourth, fifth and sixth reactors, R603, R604 and R605, respectively, during the P90117 pilot plant continuous process for the time period of from 4:00 a.m. to 13:00 (1:00 p.m.) (First Pearson Declaration, ¶22; Farris Declaration, ¶22). More specifically, area percentages of octaester set forth in Exhibit 8 are presented herein in Table 9:

**Table 9: Area Percentage of Octaester in Sucrose Esters
in R603, R604 and R605 Samples on January 20, 1989**

Time	R603	R604	R605
4:00	68.4	86.9	n/a*
5:00	n/a	n/a	92.4
6:00	67.3	85.3	n/a
7:00	n/a	n/a	94.08
8:00	65.9	86.06	n/a
9:00	n/a	n/a	93.78
10:00	65.61	86.46	n/a
11:00	n/a	n/a	92.95
12:00	67.73	83.60	n/a
13:00	n/a	n/a	95.49

*n/a = data not available

(First Pearson Declaration, ¶22; Farris Declaration, ¶22).

Exhibit 8 demonstrates octaester was present in the R603, R604 and R605 samples for the noted period of time during the P90117 pilot plant continuous process. Since, as set forth in Table 6, no octaester was present in the out-going product stream from the first reactor R600, Exhibit 8 demonstrates that the reaction mixture in the out-going product stream from reactor R600 was further reacted in subsequent reaction stages (at least in the reactors R603-R605) to the polyol fatty acid esters (sucrose octaesters) (First Pearson Declaration, ¶22; Farris Declaration, ¶22).

Therefore, the P90117 study comprised subsequent reaction stages, and the reaction mixture from the initial stage was further reacted in the subsequent reaction stages.

7. The P90117 Pilot Plant Continuous Process was Suitable for Its Intended Purpose

To show a reduction to practice of an invention, a junior party must demonstrate that the invention is suitable for its intended purpose. *Scott v. Finney*, 32 U.S.P.Q.2d 1115, 1118 (Fed. Cir. 1994). “To establish a reduction to practice of a method count, it is first necessary to show that each step of the method was performed....Second, it must be shown that the product produced by the method was satisfactory for its intended use.” *English v. Ausnit*, *supra* at 1634. Commercial perfection is not required to prove an actual reduction to practice. *English v. Ausnit*, *Id.* When an interference count does not specify any particular use, evidence which proves substantial utility for any purpose is sufficient to establish reduction to practice. *Gordon v. Hubbard*, 146 U.S.P.Q. 303, 307 (CCPA 1965).

Sucrose fatty acid polyesters have a known utility as a fat substitute in food products, as evidenced by patents such as Mattson et al, U. S. Patent No. 3,600,186 (column 2, lines 9-34); Rizzi et al, U. S. Patent No. 3,963,699 (column 1, lines 9-13); and Volpenhein, U. S. Patent No. 4,517,360 (column 1, lines 11-13), Exhibits 10, 11 and 12, respectively. The Appleby et al application indicates that preferred polyol polyesters are those which have been esterified to a level of more than about 50%, preferably more than about 70%, and more preferably more than about 80% octaester (Appleby et al application, page 29, line 36 - page 30, line 1).

As set forth in Exhibit 8, reaction samples from the first reactor, R600, indicated that the product therein comprised sucrose fatty acid mono, di, tri, tetra and pentaesters, while reaction samples from the sixth reactor, R605, indicated that the sucrose ester product comprised at least 92% sucrose fatty acid octaester, by area of total sucrose ester (First Pearson Declaration, ¶22; Farris

Declaration, ¶22). As the product formed on January 20, 1989 during the P90117 pilot plant continuous process had the desired sucrose fatty acid polyester content, including a desirable sucrose fatty acid octaester content, the product of the P90117 pilot plant continuous process was suitable for its intended purpose.

C. Party Appleby et al Have Made a *Prima Facie* Showing of Priority

Appleby et al have shown by a preponderance of evidence the completion of every essential limitation of the proposed count at least as early as January 20, 1989. Appleby et al have therefore shown by a preponderance of evidence that their invention corresponding to the proposed count was reduced to practice at least as early as January 20, 1989 and thus prior to the earliest alleged priority date of the Buter patent. Thus, Appleby et al have made a *prima facie* showing that Appleby et al are entitled to judgment relative to Buter, as required by 37 C.F.R. §1.608(b). *Hahn v. Wong, supra* at 1317 (Fed. Cir. 1989).

II. THERE WAS NO ABANDONMENT, SUPPRESSION OR CONCEALMENT OF THE APPLEBY ET AL INVENTION

35 U.S.C. § 102(g) provides that an applicant shall be entitled to a patent unless, before the applicant's invention, the invention was made in this country by another who had not abandoned, suppressed or concealed it. The time between the Appleby et al reduction to practice in late January of 1989 and the filing of the original Appleby et al patent application on September 11, 1990 does not provide any basis for inferring that the Appleby et al invention was abandoned, suppressed or concealed. Rather, the Declarations of the coinventors Corrigan, Kao, Pearson (Second Declaration) and Schafermeyer and the noninventors Steven R. Alexander, Robert B. Aylor, Roger S. Berger and Ephraim L. Kelly, and supporting exhibits thereto, establish that during the January 1989 to

September 1990 time period (less than 20 months), Appleby et al and other employees of the assignee, The Procter & Gamble Company, on their behalf were continuously involved in the research and development of various specific aspects of their complex invention and improvements relating thereto and in preparation of the Appleby et al application setting forth the invention and improvements. Thus, the time period between the actual reduction to practice and the filing of the Appleby et al patent application was reasonable, and no abandonment, suppression, or concealment of the Appleby et al invention occurred.

A. A Delay May Be Reasonable or Excused

When determining whether an inventor has abandoned, suppressed or concealed an invention, a period of delay between completion of the invention and subsequent public disclosure or the application filing may or may not be of legal consequence, and there is no particular length of delay that is *per se* unreasonable. *Checkpoint Systems v. U.S. International Trade Commission*, 54 F.3d 756, 761 (Fed. Cir. 1995). It is not the amount of time which has elapsed that is the controlling factor, but rather the total conduct of the inventor. *Fujikawa v. Wattanasin*, 39 U.S.P.Q.2d 1895, 1902 (Fed. Cir. 1996). Thus, the circumstances surrounding an inventor's delay and the reasonableness of that delay are important factors which must be considered. *Checkpoint Systems v. U.S. International Trade Commission*, *supra* at 761; *Fujikawa v. Wattanasin*, *supra* at 1902. For example, a delay may be inconsequential if it is reasonable in length, particularly where the invention is complex and/or the inventor and assignee were engaged in significant steps towards perfecting the invention and preparing an application. *Fujikawa v. Wattanasin*, *supra* at 1903. Where the inventor has been perfecting an invention, the subject matter of the improvement must

be disclosed in the patent application if the applicant is going to rely upon such activity to justify a delay in filing the patent application. *Lutzker v. Plet*, 6 U.S.P.Q.2d 1370, 1372 (Fed. Cir. 1988).

In *Fujikawa*, the Federal Circuit held that an inference of suppression or concealment was *not* warranted based upon the record as whole, where the subject matter at issue was complex, testing had been conducted during the period in question, and, throughout the 17 month delay, the assignee was moving, albeit slowly, towards filing of a patent application. *Id.* The *Fujikawa* Court also noted that while consideration of spurring (filing by a second party after disclosure by a first party) can be a factor in priority determinations, a reasonable fact finder could not have found spurring where the second party's attorney had begun work on the patent application before knowledge of the other party's patent. *Id.*

Likewise, in *Halbert v. Schuurs*, 220 U.S.P.Q. 558 (Bd. Pat. Int'f. 1983), the Board held that a 20 month delay between a reduction to practice and a patent application filing was not sufficient to raise an inference of suppression or concealment given the nature and complexity of the process involved, a method for detecting the presence of a component of an antigen-antibody reaction in a liquid sample. *Id.*, at 565. The Board noted that the inventors and assignee had also been experimenting with numerous changes to variables, such as reagents, apparatus and process conditions, during the 20 month delay period. *Id.*

Similarly, in *Bigham v. Godtfredsen*, 222 U.S.P.Q. 632 (Bd. Pat. Int'f 1984), the Board held that a 17½ month delay between an actual reduction to practice and the filing of a patent application was not inordinately long given the complexity of the invention, a pharmaceutical compound for the treatment of bacterial infections in mammals. *Id.*, at 638. The Board noted that there were numerous activities during this period relating to the subject matter in issue, including the synthesis of

compounds, tests of some of these compounds and preparation of a patent application. *See also, Rosenblum v. Hiroshima*, 219 U.S.P.Q. 844 (Bd. Pat. Int’f. 1983) (holding that a 22 month delay was reasonable where the patentee and assignee were involved in continuing engineering effort and had no intent to conceal or suppress); *Jacobs v. Lawson*, 214 U.S.P.Q. 907, 911 (Bd. Pat. Int’f. 1982) (stating that a 16 month delay was reasonable due to additional research activities); *D’Silva v. Drabek*, 214 U.S.P.Q. 556, 564 (Bd. Pat. Int’f. 1981) (holding a 17 month delay was reasonable where the inventor was working toward a more practical and direct method of preparing the inventive compound, which involved complex chemical compounds and insecticidal processes, because the public benefits from allowing inventors and their assignees to become properly acquainted with the subject matter).

As did the inventors in the aforementioned cases, the Appleby et al inventors, and others on their behalf, engaged in substantial activities from January 1989 to September 1990 directed to further research, investigation and development of their complex invention and improvements relating thereto and directed to preparation of their patent application which discloses their invention and improvements relating thereto. Accordingly, the time between Appleby et al’s reduction to practice and the filing of the Appleby et al application is reasonable, and no abandonment, suppression or concealment occurred.

B. The Appleby et al Invention is Complex

The invention disclosed in the present Appleby et al application is directed to improved synthesis of higher polyol fatty acid polyesters, especially sucrose polyesters, and more especially via transesterification reactions that do not use a solvent to form a homogeneous reaction mix, and preferably, more specifically to a continuous process for making polyol fatty acid polyesters more

efficiently and/or making polyesters of improved quality (Appleby et al application, page 1, lines 13-19).

Processes for producing polyol fatty acid esters were known prior to the Appleby et al invention. For example, Rizzi et al, U.S. Patent No. 3,963,699 (Exhibit 11), discuss a reaction for producing polyol fatty acid polyesters and exemplify a bench scale sucrose octaester batch reaction conducted in a one liter reactor (Exhibit 11, columns 5-8). Likewise, Volpenhein, U.S. Patent No. 4,518,772 (Exhibit 12), discloses a process directed to an improved solvent-free transesterification process for synthesizing polyol fatty acid polyesters. The examples described therein are bench scale sucrose octaester batch reactions performed in a one liter flask (Exhibit 12, columns 6-8). While Rizzi et al and Volpenhein et al exemplify bench-scale batch reactions for producing polyol fatty acid esters, they do not disclose how to efficiently produce polyol polyesters in a continuous process, particularly while minimizing the amount of undesirable by-products which are produced.

The polyol polyester reaction to which the Appleby et al invention is directed involves a variety of reaction parameters, for example, polyol particle size, types and levels of catalyst, soap quantities, soap removal, unreacted polyol and/or catalyst removal, ester to polyol molar ratios, reaction temperatures and pressures, plug flow conditions, and combinations of these parameters which Appleby et al have discovered influence the speed and efficiency of the transesterification reaction and the quality of the polyol polyester product (Appleby et al application, page 22, line 6- page 24, line 6). Moreover, in addition to the principal transesterification reactions, these parameters also influence side reactions, particularly in later stages of higher polyester formation, which result in undesirable by-products such as betaketo ester (BKE) and difatty ketone (DFK) in the polyol fatty

acid polyester product (Appleby et al application, page 24, lines 6-16; Kao Declaration, ¶7; Exhibit 49; Schafermeyer Declaration, ¶9; Exhibit 55).

In view of the various reaction parameters and reaction kinetics which influence the continuous process efficiency and the resulting polyol polyester product quality, the subject matter of the Appleby et al invention is complex and the period between the January 1989 reduction to practice and the September 1990 filing of the Appleby et al application in which further investigation was made is not unreasonable.

C. Appleby et al Were Engaged In Substantial Activities Between The Actual Reduction To Practice And The Filing Of The Patent Application

Moreover, from the actual reduction to practice in late January 1989 to the filing of the Appleby et al patent application in early September 1990, Appleby et al and other employees of the assignee, The Procter & Gamble Company, on their behalf were continually involved in substantial activities directed to investigation of various aspects and perfection of various improvements of their invention relating to the polyol polyester process, and particularly the sucrose polyester reaction. These activities were directed to investigating and quantifying the effects of various reaction parameters on the sucrose polyester reaction efficiency and/or the effects of the various reaction parameters on the quality of the sucrose polyester product, including the influence of various reaction parameters on the formation of undesirable DFK by-product.

Generally, from January 1989 through March 1990, a series of pilot plant runs of the continuous sucrose polyester production process were performed under the direction and control of the coinventor Pearson using the pilot plant located in Building 96 of The Procter & Gamble Company's Ivorydale plant to investigate various parameters of the continuous sucrose fatty acid

polyester process (Second Pearson Declaration, ¶2). Subsequent to each pilot plant run, the data collected during the pilot plant run was analyzed over a period of time, typically ranging from one to several weeks, in order to determine if any hypotheses and/or conclusions concerning refinement and/or improvement of the sucrose polyester reaction and/or the sucrose polyester continuous process could be made; hypotheses and/or conclusions resulting from a pilot plant run were typically investigated further in additional laboratory experiments and/or pilot plant runs (Second Pearson Declaration, ¶3). Additionally, from January 1989 through June 1990, numerous experiments were conducted by the Appleby et al coinventors and others to study the various reaction parameters. Finally, at least from June 1990 until September 1990, the Appleby et al patent application was undergoing preparation, review and revision for filing.

Summarized below, by month, are examples of the activities conducted by Appleby et al and others on their behalf between the actual reduction to practice of the invention in late January 1989 and the filing of the Appleby et al application in early September 1990. These activities demonstrate the continuous and substantial nature of the investigation of the Appleby et al invention and the preparation of the Appleby et al application during that period:

- **January 1989:** Reduced Appleby et al invention to practice; began construction of the continuous laboratory scale reactor;
- **February 1989:** Investigated residual potassium hydroxide in soap as catalyst;
- **March 1989:** Investigated sucrose particle size and milling; continued investigation of residual potassium hydroxide in soap;
- **April 1989:** Investigated sucrose particle size; investigated removal of soap/polyol/catalyst during reaction; investigated lower reaction temperatures; investigated lower temperatures in combination with higher pressures;

- **May 1989:** Continued investigation of lower reaction temperatures; investigated low levels of soap;
- **June 1989:** Continued investigation of low levels of soap and lower reaction temperatures;
- **July 1989:** Completed construction of continuous laboratory scale reactor; continued investigation of low reaction temperatures and effects on DFK;
- **August 1989:** Investigated effect of sucrose particle size in continuous laboratory scale reactor;
- **September 1989:** Continued investigation of sucrose particle size in laboratory scale continuous reactor; continued investigation of removal of soap/polyol/catalyst during reaction, investigated various ester to sucrose ratios; continued investigation of lower reaction temperatures;
- **October 1989:** Continued investigation of removal of soap/polyol/catalyst during reaction; continued investigation of various ester to sucrose ratios; continued investigation of lower reaction temperatures;
- **November 1989:** Continued investigation of various soap levels; investigated effects of varying catalyst and soap levels on DFK and I-bar;
- **December 1989:** Obtained jet mill and continued investigation of small sucrose particle sizes; investigated low temperatures in combination with removal of soap/polyol/catalyst during reaction; constructed plug flow packed column and investigated higher pressures;
- **January 1990:** Investigated lower temperatures in combination with removal of soap/polyol/catalyst during reaction; continued investigation of higher pressures and mass transfer in packed column reactor;
- **February 1990:** Investigated low ester to sucrose ratios and higher pressures; continued investigation of higher pressures and plug flow; investigated alternate catalysts;
- **March 1990:** Further investigated lower ester to sucrose ratios; submitted invention disclosure relating to ester to polyol molar ratio to the patent division; investigated higher pressures; continued investigation of lower temperatures;
- **April 1990:** Investigated ester to sucrose ratios in combination with higher pressure, gas sparging and lower temperature in later stage of the reaction;

- **May 1990:** Investigated plug flow using packed column and tray column reactors; continued investigation of alternate catalysts; continued investigation of ester to sucrose ratio;
- **June 1990:** Continued investigation of plug flow; drafted Appleby et al patent application;
- **July 1990:** Distributed drafts of the Appleby patent application for inventor review; revised drafts of the Appleby et al patent application;
- **August 1990:** Continued revision of drafts of the Appleby et al patent application;
- **September 1990:** Filed the Appleby et al patent application.

The above-referenced activities of Appleby et al and others on their behalf are described in greater detail by investigated subject matter in the following discussion, with reference to supporting evidentiary Declarations Under 37 C.F.R. §1.608 of the coinventors Corrigan, Kao, Pearson (Second), and Schafermeyer and noninventors Alexander, Aylor, Berger, Hawkins and Kelly, the supporting exhibits, and portions of the Appleby et al application where the subject matter of these activities is disclosed.

1. Polyol Particle Size

The effects of polyol particle size on the sucrose polyester reaction and the sucrose polyester product were investigated from March 1989 through December 1989, and the results of the investigations relating to polyol particle size are generally disclosed at pages 13 and 14 of the Appleby et al application.

More specifically in March 1989, a pilot plant continuous process designated P90327 investigated the effect of the use of milled sucrose particles on the sucrose polyester reaction (Second Pearson Declaration, ¶¶ 5-8; Exhibits 13 and 14). Sucrose particle sizes of 16 microns and

21 microns were tested (Second Pearson Declaration, ¶7, Exhibit 14). These sizes are within the preferred ranges disclosed in the Appleby et al application at page 13, line 38 - page 14, line 2. The P90327 pilot plant continuous process demonstrated that smaller sucrose particles were utilized more effectively in the first stage of the reaction than the larger sucrose particles, and particularly that the unreacted sucrose concentration was lower (0.37% versus 0.93%) when the smaller sucrose particles were used (Second Pearson Declaration, ¶8, Exhibit 14), thereby demonstrating a faster reaction speed was obtained using the smaller size sucrose particles, as discussed at page 13, lines 8-10 of the Appleby et al application. The conversion to octaester was greater in the reaction employing the smaller sucrose particle size (*Id.*).

In April and May 1989, a pilot plant continuous process designated P90424 further investigated the effect of milled sucrose in the sucrose polyester reaction, particularly in combination with the use of lower temperatures in later stages of the reaction (Second Pearson Declaration, ¶¶ 9-11; Exhibits 15 and 16). The P90424 continuous process demonstrated increased sucrose conversion to octaester was obtained and the amount of burnt sucrose was decreased by using a lower later stage temperature of 265°F and wet milled sucrose (*Id.*). The Appleby et al application discusses the importance of small polyol particle size at page 13, line 6 - page 14, line 8 and of lower later stage reaction temperatures at page 18, lines 1-10.

In August, September and December of 1989, experiments under the direction and control of the coinventor Corrigan using the laboratory continuous reactor system further evaluated the effect of small sucrose particle sizes (Corrigan Declaration, ¶¶ 8-10; Berger Declaration, ¶¶ 6-8 and 11-14; Exhibits 28, 30, 42 and 43). These experiments were intended to determine the effect of

sucrose particle size distributions on the continuous reaction as well as to determine the lower size limit for sucrose particles, if any (Corrigan Declaration, ¶8; Exhibit 42).

Specifically, the laboratory continuous reactor system had been constructed under the coinventor Corrigan's direction and control from January to July 1989 and was ideally suited for these tests because it required only a small amount of sucrose (e.g., 2 kg) such that the sucrose could be easily sieved to obtain the desired size distributions (Corrigan Declaration, ¶¶ 6 and 8, Exhibits 39, 40 and 42). In September 1989, sucrose particles having average particle sizes less than about 63 microns and less than about 38 microns, respectively, were tested using the laboratory continuous reactor system. The reaction employing the smaller sucrose particles achieved a higher average degree of esterification, I-bar of 2.97 using the less than about 38 micron sucrose particles versus I-bar of 2.77 using the less than about 63 micron sucrose particles, and the greater sucrose utilization, 3% unreacted sucrose at 4.5 hours using the less than about 38 micron sucrose particles versus 5% unreacted sucrose at 4.5 hours using the less than about 63 micron sucrose particles (Corrigan Declaration, ¶9; Berger Declaration, ¶¶ 6-8; Exhibit 28). These results demonstrated faster and more efficient reactions were obtained using smaller polyol particles as discussed in the Appleby et al application at page 13, line 6 - page 14, line 4 (Corrigan Declaration, ¶9).

In December 1989, jet milling of sucrose was investigated as a means for producing sucrose having an average particle size less than 10 microns (Corrigan Declaration, ¶10; Berger Declaration, ¶¶ 11-14, Exhibit 30). A jet mill was installed for in-house production of sucrose particles of about 10 microns or less (Corrigan Declaration, ¶10; Berger Declaration, ¶12; Exhibit 30). Sucrose polyester reactions using the jet-milled sucrose were performed and resulted in sucrose polyester products having equivalent or increased average degrees of esterification and lower amounts of

unreacted sucrose than the previous September 1989 tests which used larger sized sucrose particles (Corrigan Declaration, ¶¶ 9-10; Berger Declaration ¶¶ 12-14; Exhibits 28 and 30). These results demonstrated that the sucrose polyol particle sizes within the ranges disclosed at page 14, lines 1 - 4 of the Appleby et al application increased reaction speed and efficiency as disclosed at page 13, lines 8-10 of the Appleby et al application (Corrigan Declaration, ¶10).

2. Types and Levels of Catalyst

The effects of using various types of catalyst and varying quantities of catalyst on the sucrose polyester reaction and the sucrose polyester product were investigated in February-April and November 1989 and in February and May 1990. The results of the investigations relating to catalyst types and quantities are generally disclosed at pages 14 and 15 of the Appleby et al application.

More specifically, in February 1989 the coinventor Schafermeyer speculated that residual potassium hydroxide (KOH) contained in the potassium soap emulsifier which was used in the sucrose polyester reaction acted as a hidden catalyst for the sucrose polyester reaction (Schafermeyer Declaration, ¶7, Exhibit 54). In February and early March 1989, the coinventor Kao noted that high levels of KOH in the soap resulted in fast first or initial stage reactions and poor sucrose utilization while low levels of KOH resulted in significantly better sucrose utilization (Kao Declaration, ¶¶ 8-9; Exhibit 50), thereby demonstrating that KOH base which accompanies the soap effects the sucrose polyester reaction as set forth in the Appleby et al application at page 14, lines 23-25. Additional sucrose polyester experiments were performed in March and April 1989 under the direction and control of the coinventor Schafermeyer and employed residual potassium hydroxide in the soap as the only catalyst in the reaction, i.e., potassium carbonate catalyst was not added to the reaction (Schafermeyer Declaration, ¶8; Kelly Declaration, ¶¶ 5-8; Exhibit 35). These reactions produced

sucrose polyester product having high octaester content and high average degrees of esterification, thereby demonstrating that the small amount of the base that typically accompanies the soap can be a preferred catalyst, as disclosed at page 14, lines 15-27 of the Appleby et al application.

In November 1989, experiments were conducted to investigate, inter alia, what effect varying the amount of catalyst employed in the sucrose polyester reaction might have on the DFK content and the I-bar of the sucrose ester product (Berger Declaration, ¶¶ 9-10; Exhibit 29). As set forth at page 14, lines 12-15 of the Appleby et al application, low levels of catalyst provide fast reaction with little formation of undesirable materials.

In February and May 1990, the use of alternate catalysts such as sodium methoxide and potassium methoxide in the sucrose polyester reaction was investigated under the direction and control of the coinventor Corrigan (Corrigan Declaration, ¶14; Berger Declaration, ¶¶ 22-29; Exhibit 32). These alternate catalysts were added at a later stage of the reaction and produced an I-bar value of greater than 7, thereby demonstrating the feasibility of using these alternate catalysts in a later stage of the reaction, as generally disclosed at page 14, lines 27-32 of the Appleby et al application.

3. Amount of Soap

The effect of the amount of soap emulsifier employed in the sucrose polyester process was investigated in May, June and November 1989 and the results of these investigations are generally disclosed at page 15 of the Appleby et al application.

More specifically, in May 1989, attention turned to the amount of soap employed in the sucrose polyester reaction and a matrix of experiments was performed to investigate the effect of various soap levels on the sucrose polyester reaction (Corrigan Declaration, ¶7; Kelly Declaration, ¶¶ 9-12; Exhibits 36 and 41). Soap to polyol molar ratios of 0.35:1, 0.42:1, and 0.44:1 were

employed, these values being within the preferred ranges disclosed in the Appleby et al application at page 15, lines 15-19 (Corrigan Declaration, ¶7; Kelly Declaration, ¶¶ 10-12; Exhibit 36). Sucrose polyester product comprising 97% or greater octaester was obtained using these low soap levels (*Id.*), thereby demonstrating that low levels of soap could be used in the sucrose polyester reaction, as disclosed at page 15, lines 4-10 of the Appleby et al application.

In June 1989, a pilot plant continuous process designated P90605 investigated the effect of various soap levels on the sucrose polyester continuous reaction in combination with the use of milled sucrose and lower later stage temperatures (Second Pearson Declaration, ¶¶12-17, Exhibits 17-19). Soap to polyol molar ratios of 1.18:1 and 0.59:1 were employed in respective runs, the second of which values is within the range disclosed in the Appleby et al application at page 15, lines 15-17, and a "no soap" run was also performed (Second Pearson Declaration, ¶14; Exhibit 17). The P90605 pilot plant continuous process demonstrated that the amount of soap present in the reaction had a strong effect on the reaction rate (Second Pearson Declaration, ¶16, Exhibit 18).

In November 1989, experiments were conducted to investigate, inter alia, what effect varying the amount of soap employed in the sucrose polyester reaction might have on the DFK content and the I-bar of the sucrose ester product (Berger Declaration, ¶¶ 9-10; Exhibit 29). As set forth at page 15, lines 3-6 of the Appleby et al application, low levels of soap are desirable.

4. Removal of Soap, Unreacted Polyol and/or Catalyst

The effects of removing soap, unreacted polyol and/or catalyst on the sucrose polyester process and the sucrose polyester product were investigated in April 1989 and from September 1989 through February 1990. The results of the investigations relating to removal of soap, unreacted polyol and/or catalyst are generally disclosed at pages 15 and 16 of the Appleby et al application.

More specifically, in April 1989, sucrose polyester experiments were performed wherein the reaction mixture was filtered to remove unreacted sucrose when the unreacted sucrose level dropped to a concentration between about 1% and about 2% (Second Pearson Declaration, ¶29; Exhibit 25). The filtered reaction mixture contained sucrose ester product comprising about 95% octaester in 2 hours while an unfiltered control reaction mixture contained sucrose ester product comprising about 88% octaester product in 4 hours, thereby demonstrating faster reaction kinetics and highly esterified product were obtained in the filtered reaction mixture, in accordance with the teachings at page 16, line 10 - page 17, line 6 of the Appleby et al application (*Id.*).

Additional sucrose polyester experiments employing filtration were performed under the direction and control of the coinventor Kao in September and October of 1989 on the continuous laboratory scale reactor (Kao Declaration, ¶¶ 14-16; Alexander Declaration, ¶¶ 5-9, Exhibits 46, 52 and 53). In these experiments, the reaction mixture was filtered during the reaction to remove soap, unreacted and burnt sucrose and carbonate solids (Kao Declaration, ¶16; Alexander Declaration, ¶6; Exhibits 46 and 53). In one experiment, as an example, the reaction mixture was filtered when the sucrose polyester therein had an I-bar of 4.6, corresponding to an average degree of esterification of 57%, which is within the preferred ranges disclosed at page 16, lines 12-17 of the Appleby et al application (Kao Declaration, ¶15; Alexander Declaration, ¶¶ 6-7; Exhibit 46). The filtered reaction mixture comprised about 0.05% unreacted sucrose, which is within the preferred ranges disclosed at page 16, lines 35-38 of the Appleby Application (*Id.*). In a similar experiment, sucrose ester product in a filtered reaction mixture comprised about 98% octaester about 2 hours after filtration while the unfiltered control reaction mixtures contained sucrose ester comprising about 77% octaester at that same time (Alexander Declaration, ¶¶ 8-9, Exhibit 46). Thus, the filtered reaction

mixtures reacted faster and reached a high degree of esterification more quickly than the unfiltered reaction mixtures, as discussed generally at page 16, lines 12-38 of the Appleby et al application.

In December 1989 and January 1990, additional polyol fatty acid polyester experiments investigated the effects of filtration of the reaction mixture (Alexander Declaration, ¶¶ 10-16; Exhibit 47). In various experiments, the reaction mixture was filtered, for example, at I-bars of about 2.91, 1.95 and 1.90, which correspond to average degrees of esterification of about 36%, 24% and 24%, respectively (Alexander Declaration, ¶¶ 11-12, 13-14 and 15-16, respectively; Exhibit 47). In each case, the sucrose ester product in the filtered reaction mixture comprised a higher percentage of octaester and had a greater average degree of esterification than the sucrose ester product in a corresponding non-filtered control reaction (Alexander Declaration, ¶¶ 12, 14, 16; Exhibit 47). After filtering, the reaction mixtures comprised unreacted sucrose contents of 0.19%, 0.71% and 0.73%, respectively, these values being within the preferred ranges disclosed in the Appleby et al application at page 16, lines 35-38 (*Id.*).

5. Low Ester to Polyol Molar Ratios

The effects of the molar ratio of fatty acid lower alkyl ester to polyol, and particularly, the effects of low ester to polyol molar ratios, on the sucrose polyester process and the sucrose polyester product were investigated during September and October 1989 and February through May 1990. The results of the investigations relating to low ester to polyol molar ratios are generally disclosed at page 17 of the Appleby et al application.

More specifically, in September and October 1989, a matrix of polyol fatty acid polyester experiments investigated various ester to polyol molar ratios (Corrigan Declaration, ¶11; Kelly Declaration, ¶¶ 15-20; Exhibit 38). Molar ratios of total ester reactant to esterifiable sites on the

polyol of about 1:1 and about 1.16:1 were tested (Corrigan Declaration, ¶11; Kelly Declaration, ¶¶ 16, 18-20 (1:1 molar ratio), ¶17 (1.16:1 molar ratio); Exhibit 38). These ratios are within the preferred ranges disclosed at page 17, lines 8-11 of the Appleby et al application. The reactions provided sucrose ester products comprising 74% or greater octaester and therefore demonstrated that low ester to polyol ratios can be effectively used as disclosed generally at page 17, lines 7-37 of the Appleby et al application.

In February 1990, a pilot plant continuous process designated P00205 investigated, inter alia, the use of lower fatty acid methyl ester to sucrose molar ratios in a continuous reaction (Second Pearson Declaration, ¶¶ 21-23 and 27; Exhibits 22 and 24). Molar ratios of ester to esterifiable sites on the polyol of about 1.75:1, about 1.5:1, about 1.25:1 and about 1.06:1 were tested, the latter two molar ratios being within the ranges disclosed at page 17, lines 8-10 of the Appleby et al application (Second Pearson Declaration, ¶¶ 22 and 27; Exhibits 22 and 24).

In late March and early April 1990, a pilot plant continuous process designated P00326 further investigated, inter alia, the use of low fatty acid methyl ester to sucrose molar ratios in combination with lower later stage temperatures, higher pressures and gas sparging (Second Pearson Declaration, ¶¶ 24-27; Exhibits 23 and 24). Molar ratios of ester to esterifiable sites on the polyol of about 1.125:1 and about 1.7:1 were tested (Second Pearson Declaration, ¶¶ 26-27; Exhibits 23 and 24).

Sucrose ester products comprising greater than 70% octaester were obtained in the P00205 and P00326 pilot plant continuous processes using ester to esterifiable site molar ratios of about 1.25:1, 1.125:1 and 1.06:1 (Second Pearson Declaration, ¶27; Exhibit 24), all of which are within the ranges disclosed at page 17, lines 1-11 of the Appleby et al application. Additionally, the use

of gas sparging in a reaction employing a molar ratio of ester to polyol esterifiable sites of 1.125:1 improved the reaction rate, as disclosed at page 17, lines 19-29 of the Appleby et al application (*Id.*).

In March 1990, Development Record No. 7988 directed to the use of low fatty acid methyl ester to sucrose molar ratios in the sucrose polyester reaction was submitted to the Patent Division of the assignee by the coinventor Corrigan (Corrigan Declaration, ¶15; Aylor Declaration, ¶3; Exhibit 45). The subject matter of Development Record No. 7988 was incorporated into the Appleby et al application (Aylor Declaration, ¶ 4), for example at page 17, lines 7-37.

Additional experiments intended to evaluate, inter alia, the use of low molar ratios of ester to polyol esterifiable sites (1.3:1) in combination with powdered or granular sucrose and catalyst were conducted in May 1990 (Corrigan Declaration, ¶17; Berger Declaration, ¶¶ 32-34; Exhibit 34). The experiments yielded sucrose esters having a high average degree of esterification (\bar{I} of about 7.8), confirming the feasibility of using low ratios of ester to polyol esterifiable sites, as disclosed in the Appleby et al application at page 17, lines 8-11 and page 22, lines 18-20 (*Id.*).

6. Lower Reaction Temperatures

The effects of lower reaction temperatures on the sucrose polyester process and the sucrose polyester product were investigated from April 1989 through March 1990. The results of these investigations relating to the use of lower reaction temperatures are generally disclosed at pages 18 and 23 of the Appleby et al application.

Pilot plant runs were conducted to investigate, inter alia, the effect of lower later stage reaction temperatures in a pilot plant continuous reaction. In late April and early May of 1989, a pilot plant continuous process designated P90424 investigated the effect of using later stage temperatures of about 265°F and 285°F (Second Pearson Declaration, ¶¶ 9-11; Exhibit 15). The use

of a later stage reaction temperature of 265°F resulted in increased conversion of sucrose to sucrose octaester and visibly decreased amounts of burnt sucrose as compared with the use of a reaction temperature of 285°F (Second Pearson Declaration, ¶11; Exhibit 16). The lower temperature is within the range disclosed at page 18, lines 6-10 of the Appleby et al application. In June 1989, the P90605 pilot plant continuous process further investigated the effect of lower later stage reaction temperatures of about 250°F and 265°F, these values also being within the preferred ranges disclosed in the Appleby et al application at page 18, lines 7-9 (Second Pearson Declaration, ¶¶ 12, 15 and 17; Exhibits 17 and 19). For example, one run of the P90605 continuous process employed a reduced soap level in combination with a later stage reaction temperature of about 250°F and resulted in a sucrose ester product comprising about 95% octaester (Second Pearson Declaration; ¶17; Exhibit 19).

The effects of lower later stage reaction temperatures on reducing DFK formation were also under investigation. More specifically, in May and June 1989, sucrose polyester experimentation by the coinventor Kao investigated the use of lower reaction temperatures and any resulting influence on the formation of DFK ketones (Kao Declaration, ¶¶ 10-12; Exhibit 51). Sucrose polyester reactions employing later stage temperatures of 100° to about 120°C, as disclosed at page 18, lines 1-10 of the Appleby et al application, yielded sucrose ester products comprising about 80-91% octaester with DFK levels of about 87-374 ppm, which is substantially within the preferred ranges disclosed in the Appleby et al application at page 24, line 15 (Kao Declaration, ¶¶ 11-12; Exhibit 51). Further experiments were also conducted in May 1989 to investigate, inter alia, the effect of using a temperature of 100°C in a later stage of the sucrose polyester reaction; these experiments resulted in sucrose ester product comprising about 217-234 ppm DFK (Kelly

Declaration, ¶¶ 13-14; Exhibit 37). It was concluded that low temperatures are preferred for reducing DFK levels in polyol polyester product, as is set forth in the Appleby et al application at page 18, lines 6-10 and page 24, lines 1-5 (Schafermeyer Declaration, ¶¶ 10-12; Exhibits 56-58).

In September and October of 1989, a pilot plant continuous process designated P90925 investigated the use of lower later stage reaction temperatures of 220°F and 250°F, which are within the preferred ranges disclosed at page 18, lines 6-10 of the Appleby et al application (Second Pearson Declaration, ¶¶ 18-20; Exhibits 20 and 21). The use of reduced soap levels in combination with a later stage reaction temperature of 220°F resulted in increased reaction rates and high octaester conversions (Second Pearson Declaration, ¶20; Exhibit 21).

Additionally, between December 1989 and February 1990, a matrix of experiments was conducted to investigate, inter alia, the effect of later stage reaction temperatures of from about 250°F to about 325°F in combination with filtration of the reaction mixture during the reaction (Alexander Declaration, ¶¶ 10-16; Exhibit 47). An experiment employing filtration and a later stage temperature of 250°F produced a lower amount of DFK (165 ppm) than a similar experiment employing a later stage temperature of 325°F (320 ppm), demonstrating that lower amounts of DFK were produced when the later stage reaction temperature was reduced, in accordance with the teachings of the Appleby et al application at page 18, lines 9-10 which state that lower temperatures are desirable in later stages of the polyol polyester reaction to minimize side reactions (Alexander Declaration, ¶¶ 13-14, 15-16; Exhibit 47).

In March 1990, additional laboratory experiments investigated the effect of lower later stage temperatures of about 110°C in combination with higher pressures of about 30 mm Hg, in accordance with the teachings at page 23, lines 7-9 of the Appleby et al application (Corrigan

Declaration, ¶16; Berger Declaration, ¶¶ 30-31; Exhibit 33). The DFK level in the sucrose ester product was 201 ppm at the end of the reaction (Berger Declaration ¶31; Exhibit 33), and therefore within the range disclosed in the Appleby et al application at page 24, lines 13-15.

7. Higher Reaction Pressures and Plug Flow Conditions

The effects of using higher reaction pressures and plug flow conditions on the sucrose polyester process and the sucrose polyester product were investigated from December 1989 through June 1990. The results of the investigations relating to the use of higher reaction pressures and plug flow conditions are generally disclosed at pages 18 and 21 of the Appleby et al application.

In December 1989 and January 1990, a plug flow reactor comprising a packed column was constructed for investigating, inter alia, the effect of higher pressures and methanol mass transfer in later stages of the sucrose polyester reaction (Second Pearson Declaration, ¶30; Exhibit 26). A reaction using pressures of 25 mm Hg at the top of the reactor and 45 mm Hg at the bottom of the reactor provided sucrose ester product comprising about 99% octaester in less than about 2 hours, thereby demonstrating the feasibility of reacting at higher pressures while maintaining the reaction speed through more efficient means of methanol removal (*Id.*). The use of a packed column reactor in a later stage of the polyol polyester reaction is disclosed at page 21, lines 30-37 of the Appleby et al application. Reaction pressures between 15 mm Hg and 100 mm Hg at the top of the packed column reactor were also investigated (Second Pearson Declaration, ¶31; Hawkins Declaration, ¶¶ 10-12; Exhibit 27). The use of higher pressures and benefits which may be achieved through the use of packed column reactors are disclosed at page 18, lines 17-19 and page 21, lines 25-37, respectively, of the Appleby et al application.

Higher later stage reaction pressures in the sucrose polyester reaction were further investigated in February 1990. Experiments demonstrated that high octaester conversions (sucrose ester product comprising about 90% octaester) could be achieved at reaction pressures of 15 mm Hg and 25 mm Hg with nitrogen sparging (Corrigan Declaration, ¶12; Berger Declaration, ¶¶ 15-19; Exhibit 31). In March 1990, experiments using later stage pressures of 30 mm Hg and 45 mm Hg with nitrogen sparging produced sucrose ester comprising greater than 70% octaester (Corrigan Declaration, ¶12; Berger Declaration, ¶¶ 20-21; Exhibit 31). The higher pressures investigated during February and March 1990 are within the preferred ranges disclosed at page 18, lines 16-36 of the Appleby et al application (Corrigan Declaration, ¶12), and these experiments resulted in products comprising about 300 ppm or less DFK, in accordance with preferred ranges set forth in the Appleby et al application at page 24, lines 13-15 (Corrigan Declaration, ¶13; Berger Declaration, ¶¶ 16-21; Exhibits 31 and 44).

In February and March 1990, the P00205 and P00326 pilot plant continuous processes also investigated the effect of using higher later stage reaction pressures of 20 mm Hg and 60 mm Hg, respectively (Second Pearson Declaration, ¶¶ 23 and 26; Exhibits 22 and 23). During the P00205 process, a run at 20 mm Hg using a nitrogen sparge rate of 0.8 lb/hr resulted in sucrose ester product comprising about 85% octaester in about 12 hours, while a run during the P00326 process at a pressure of 60 mm Hg using a nitrogen sparge rate of 2.4 lb/hr resulted in sucrose ester comprising about 80% octaester in about 12 hours (Second Pearson Declaration, ¶28; Exhibit 24). These reactions demonstrated that higher reaction pressures could be used in combination with increased sparging rates to provide acceptable reaction speeds, as disclosed at page 18, lines 17-29 of the Appleby et al application (*Id.*).

In April, May and June 1990, additional experiments using a packed column reactor and a fifteen tray column reactor, respectively, during a later stage of the sucrose polyester reaction were performed with the goal of producing a product having about 75% octaester using lower temperatures and higher pressures (Alexander Declaration, ¶¶ 17-24; Exhibit 48). For example, in an experiment using an initial ester to polyol esterifiable sites molar ratio of about 1.06:1, the top of the packed column reactor was operated at a pressure of from about 50-51 mm Hg and a temperature of from about 265°F to about 280°F (from about 130°C to about 138°C), the middle of the column was operated at a temperature of from about 125°C to about 110°C, and the bottom of the column was operated at a temperature of from about 109°C to about 81°C and a pressure of from about 68 mm Hg to about 72 mm Hg. A product comprising about 73% octaester was obtained (Alexander Declaration, ¶¶ 19-20; Exhibit 48). An experiment using a fifteen tray column reactor produced similar results (Alexander Declaration, ¶¶ 22-24; Exhibit 48). These experiments demonstrated that relatively high octaester conversions can be achieved with plug flow conditions in a later stage of the reaction as described in the Appleby et al application at page 21, lines 25-27.

8. Pilot Plant Runs Employing Combinations of Parameters

Various combinations of reaction parameters as disclosed at pages 22 and 23 of the Appleby et al application were investigated in the pilot plant continuous processes conducted between the January 1989 actual reduction to practice and the September 1990 filing of the Appleby et al application. While these pilot plant runs have been referred to above with respect to individual parameters, the combinations of parameters studied during the respective pilot plant continuous processes described in the Second Pearson Declaration are summarized in Table 10:

**Table 10: Pilot Plant Continuous Processes
Investigating Combinations of Parameters**

Pilot Plant Continuous Process	Combination of Improvements Tested	Date	¶¶ Referenced in Second Pearson Declaration
P90424	reduced polyol particle sizes, reduced later stage temperatures	April 1989	9-11
P90605	reduced soap levels; reduced later stage temperatures	June 1989	12-17
P90925	reduced soap levels; reduced later stage temperatures	September 1989	18-20
P00205	increased pressures and decreased temperatures in later reaction stage; reduced ester to polyol ratios; decreased later stage temperature	February 1990	21-23 and 27-28
P00326	reduced ester to sucrose ratios; reduced later stage temperatures with increased sparging rate; increased later stage pressures with increased sparging rate	March 1990	24-28

Subsequent to each pilot plant continuous process, the data collected during the process was analyzed by Mr. Pearson and others over a period of time, typically ranging from one to several weeks, in order to determine if any hypothesis and/or conclusions concerning refinement and/or improvement of the sucrose polyester reaction and/or the sucrose polyester continuous process could be made; hypotheses and/or conclusions resulting from a pilot plant run were typically investigated further in additional laboratory and/or pilot plant runs (Second Pearson Declaration, ¶3).

9. Preparation of the Appleby et al Patent Application

A significant amount of time was also devoted to the preparation of the Appleby et al patent application during the time between the actual reduction to practice in January 1989 and the filing of the Appleby et al application in September 1990. For example, Mr. Robert Aylor, a patent attorney employed in the Patent Division of The Procter & Gamble Company, had been assigned the task of preparing the Appleby et al application. Prior to July 3, 1990, he began preparing a patent application which incorporated the substance of the 7988 Development Record submitted to the Patent Division by the coinventor Corrigan in March 1990, as well as other aspects of the sucrose polyester process then under development by the Appleby et al inventors (Aylor Declaration, ¶¶ 3-4). This patent application, entitled "Polyester Polyol Synthesis," was filed on September 11, 1990 and assigned Serial No. 07/580,706 (hereinafter the Appleby et al '706 Application) and is a parent patent application for the present Appleby et al U.S. Application Serial No. 08/360,184 (Aylor Declaration, ¶4).

On or about July 3, 1990, Mr. Aylor forwarded a draft of the Appleby et al application to the coinventor David J. Bruno for review by the inventors (Aylor Declaration, ¶5; Exhibit 59). On or about July 18, 1990, Mr. Aylor forwarded a revised draft of the Appleby et al application to the coinventor Corrigan for review by the inventors (Aylor Declaration, ¶6; Exhibit 60). Between July 18, 1990 and the September 11, 1990 filing date of the Appleby et al application, Mr. Aylor further revised the application and incorporated additional information from the Appleby et al inventors into the application (Aylor Declaration, ¶7). Thus, the preparation of the Appleby et al patent application extended at least from June 1990 through to the September 1990 filing date.

The Buter patent which is the subject of the concurrently filed Amendment and Request for Interference Under 37 C.F.R. §1.607 claims priority from European Patent Applications Nos. 89200371.6 filed on February 16, 1989 (the '371 European Application) and 89202931.5 filed November 20, 1989 (the '931 European Application). The '371 and '931 European Patent Applications also provide priority to European Patent Application No. 90200336.7 filed February 14, 1990 (the '336 European Application), which published on August 22, 1990, as evidenced by the European A1 Publication No. 383,404 A1, a copy of which is provided as Exhibit 61. Mr. Aylor did not become aware of the published European A1 Publication No. 383,404 A1 until subsequent to its August 22, 1990 publication date, which was well after he began preparing the Appleby et al application and after the respective drafts were sent to Mr. Bruno on or about July 3, 1990 and Mr. Corrigan on or about July 18, 1990 (Aylor Declaration, ¶8).

It is therefore clear that Mr. Aylor had not only begun work on the Appleby et al application but had prepared a substantial portion of the application prior to publication of the Buter European A1 Publication No. 383,404 A1 and prior to any knowledge by Mr. Aylor of the published application. Accordingly, there was no spurring of Appleby et al by Buter as a reasonable fact finder cannot find spurring where the second party's attorney had begun work on the patent application before knowledge of the other party's patent, *Fujikawa v. Wattanasin*, *supra* at 1903.

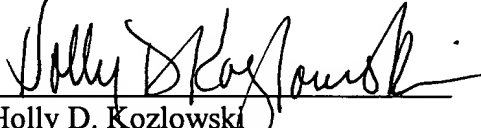
Thus, during the time between the actual reduction to practice in January 1989 and the filing of the Appleby et al application in September 1990, Appleby et al and others on their behalf were continuously involved in the research and development of various specific aspects of their complex invention and improvements relating thereto and in preparation of the Appleby et al application setting forth the invention and improvements. The time period between the actual reduction to

practice and the filing of the Appleby et al patent application was therefore reasonable, and no abandonment, suppression, or concealment of the Appleby et al invention occurred.

III. CONCLUSION

The evidence submitted herewith sets forth a factual description of acts and circumstances performed or observed by the Declarants which collectively establish by a preponderance of evidence that Appleby et al reduced to practice the invention corresponding to the count set forth in the accompanying Amendment and Request for Interference pursuant to 37 C.F.R. §1.607 at least as early as January 20, 1989, and that the Appleby et al invention was not abandoned, suppressed or concealed from January 1989 to the filing of the Appleby et al original application on September 11, 1990. Accordingly, Appleby et al are *prima facie* entitled to a judgement on priority with respect to either the Buter patent actual U.S. filing date of February 9, 1990 or either of the Buter patent's alleged effective U.S. filing dates under 35 U.S.C. §119 of February 16, 1989 or November 20, 1989. The declaration of an interference as requested in the accompanying Amendment and Request for Interference Pursuant to 37 C.F.R. §1.607 is therefore warranted.

Respectfully submitted,


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